

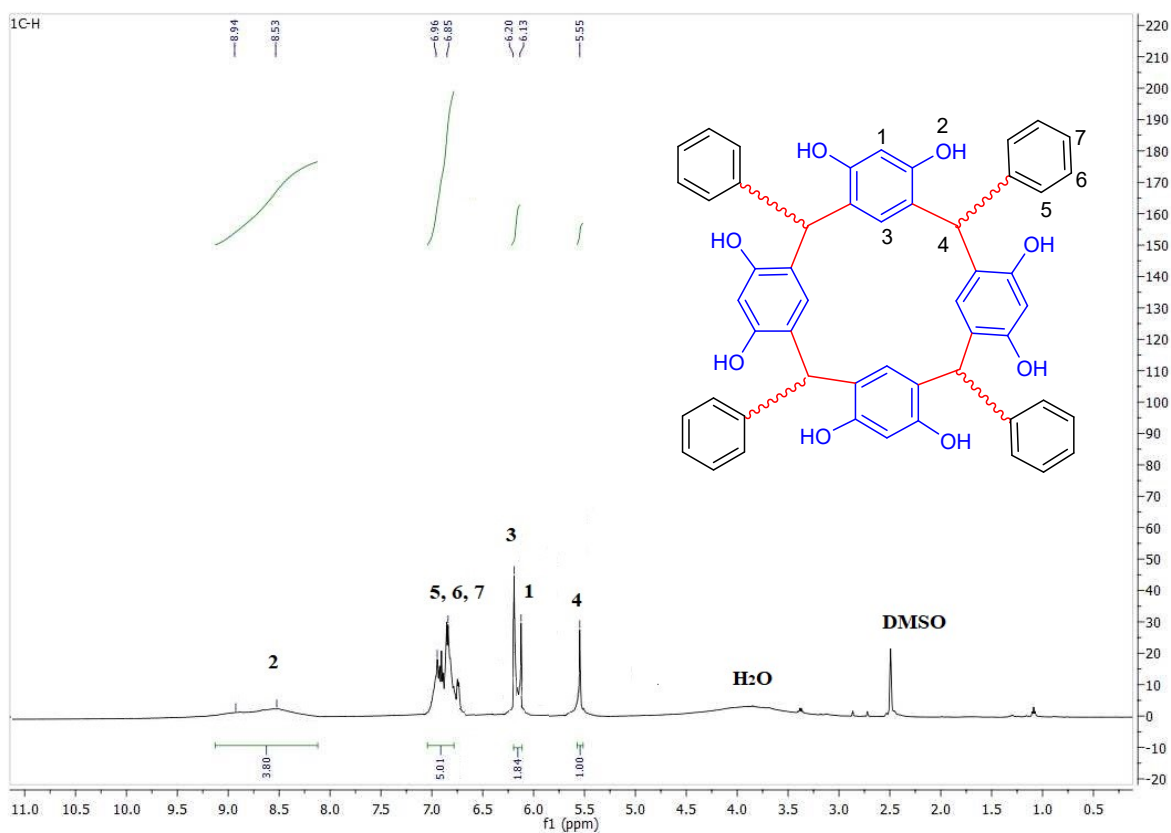
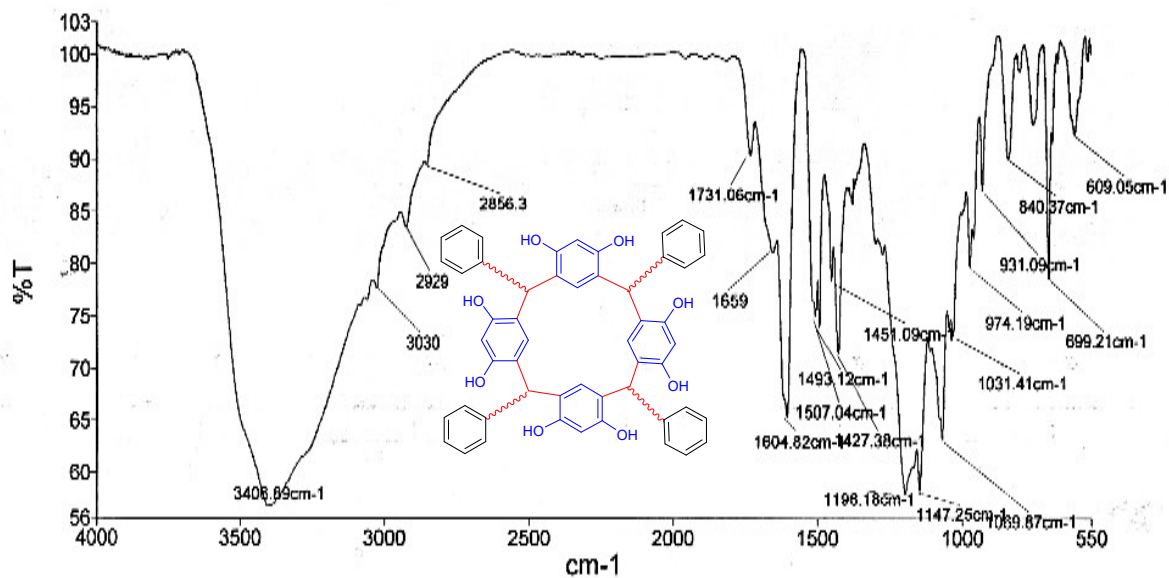
Supplementary Information

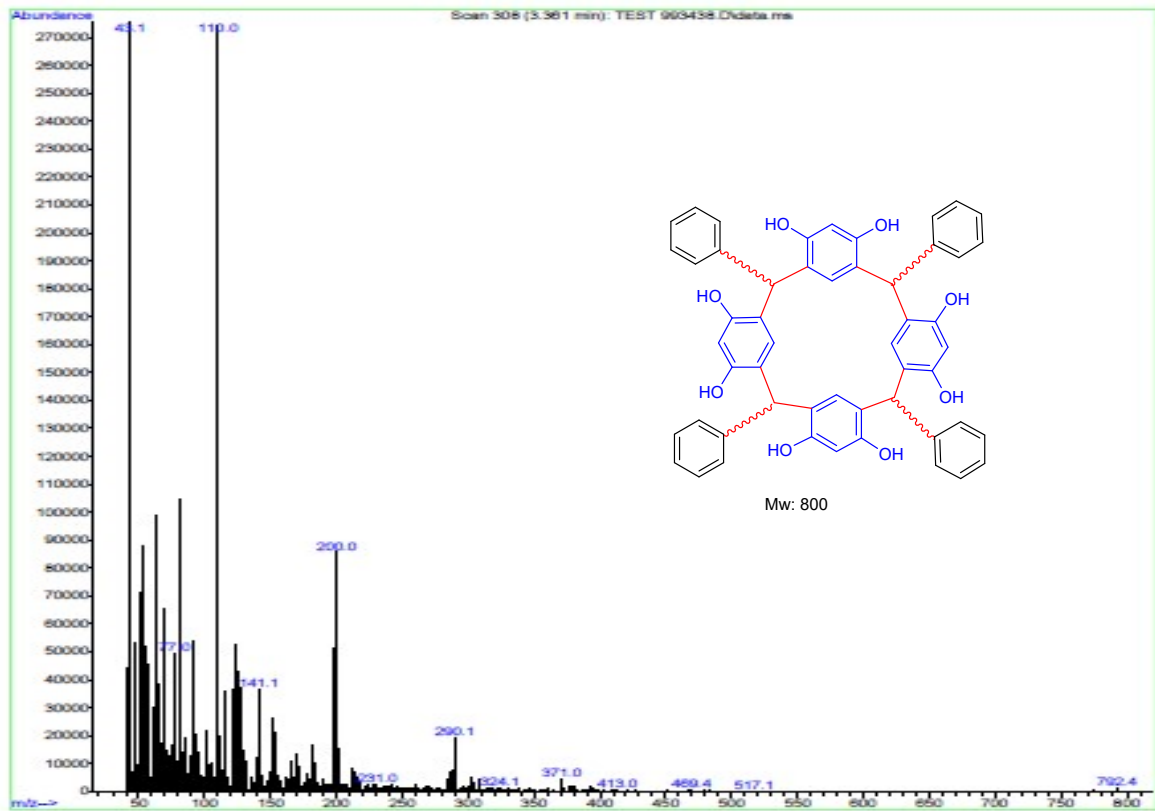
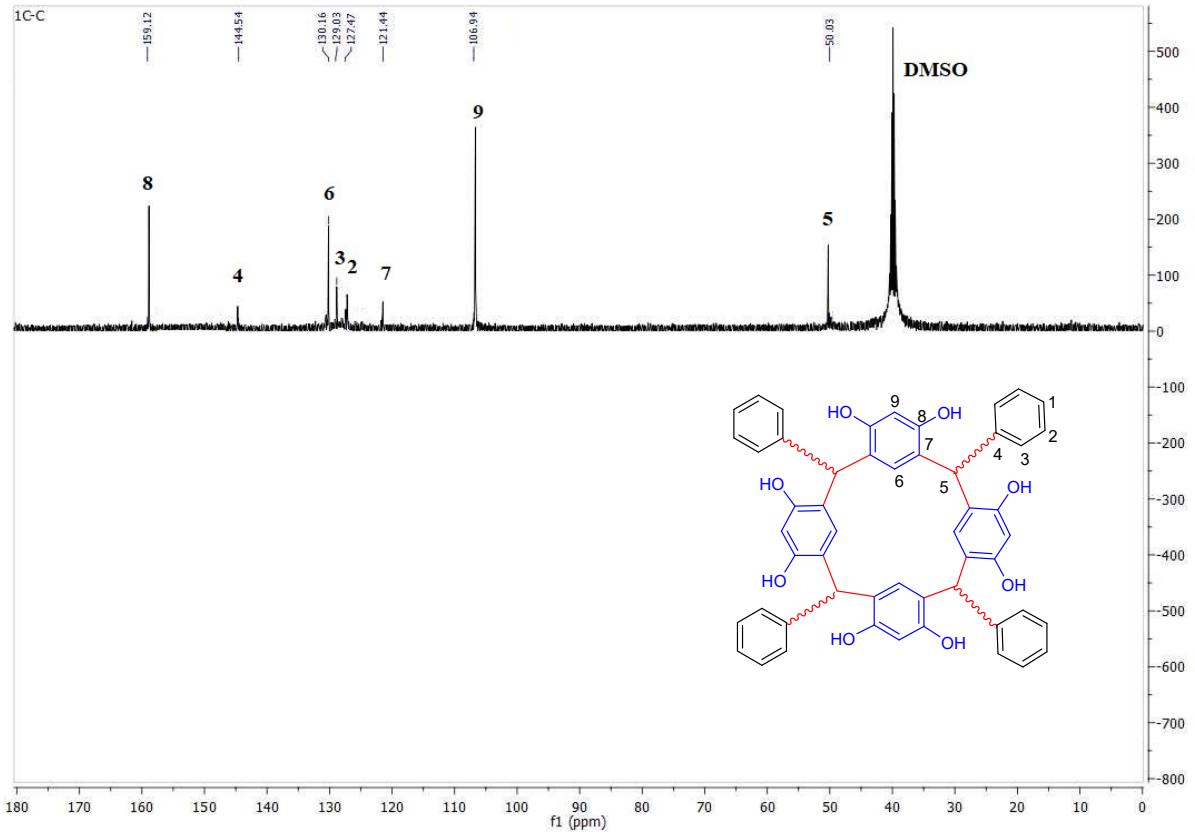
Synthesis of calixresorcarenes using magnetic poly triazine-benzene sulfonamide-SO₃H

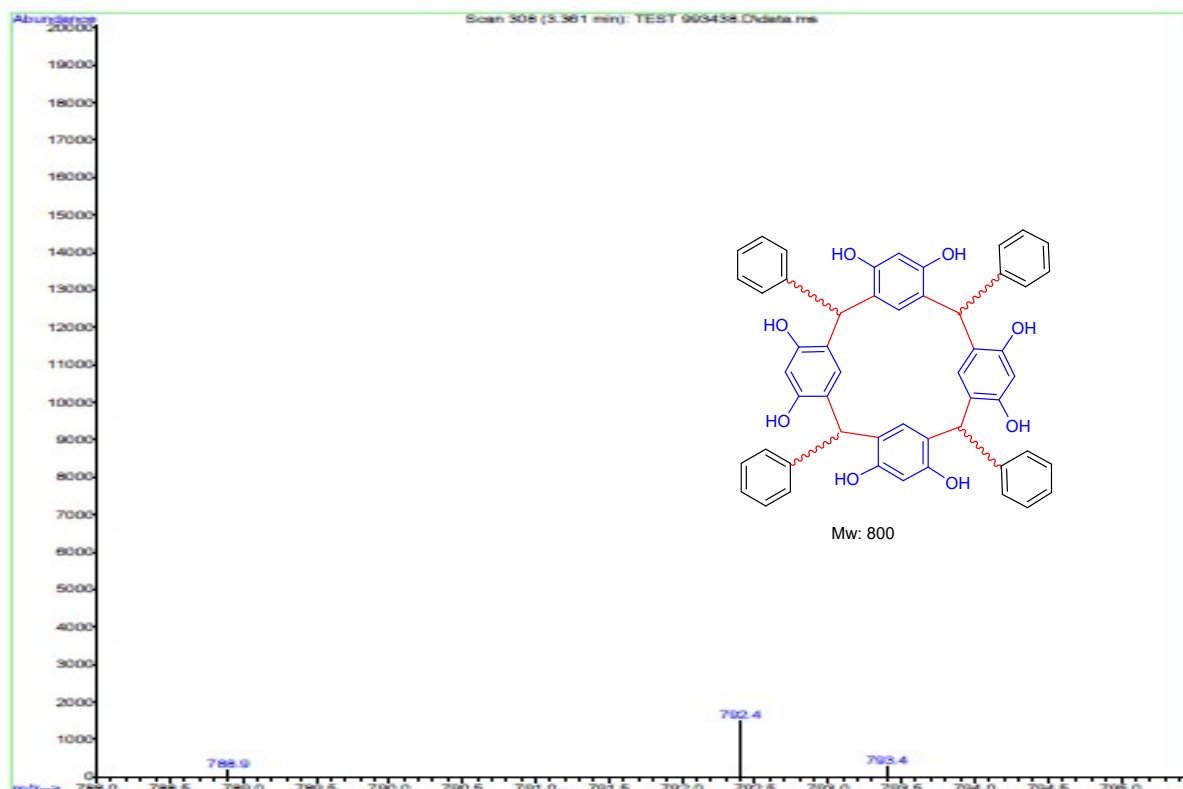
Alireza Gharekhani, Ramin Ghorbani-vaghei,* Sedigheh Alavinia
Department of Organic Chemistry, Faculty of Chemistry, Bu-Ali Sina University, 65178/38695, Hamedan, Iran.
E-mail: rgvaghei@yahoo.com or ghorbani@basu.ac.ir

2,8,14,20-Tetraphenyl-4,6,10,12,16,18,22,24-octahydroxycalix[4]-resorcarene (3a)

Colorless solid, mp > 300 °C (dec); FT-IR (KBr) ν : 3406, 3030, 2929 cm^{-1} ; ^1H NMR (500 MHz, $\text{DMSO-}d_6$) δ ppm: 5.55 (s, 1H, CH, H_4), 6.13 (s, 1H, Ar-H, H_1), 6.20 (s, 1H, Ar-H, H_3), 6.75-6.96 (m, 5H, Ar-H, $\text{H}_5, \text{H}_6, \text{H}_7$), 8.53-8.94 (OH, broad peak, H_2 , 3H), ppm. ^{13}C NMR (125 MHz, $\text{DMSO-}d_6$) δ ppm: 50.03, 106.94, 121.44, 127.44, 127.47, 129.03, 130.16, 144.54, 159.12. MS: $m/z = 702$ [M^+].

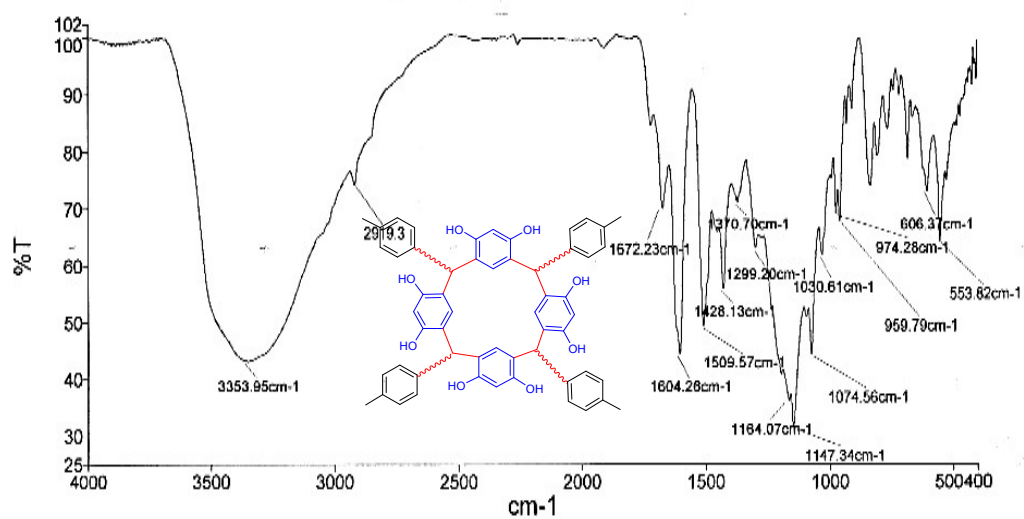


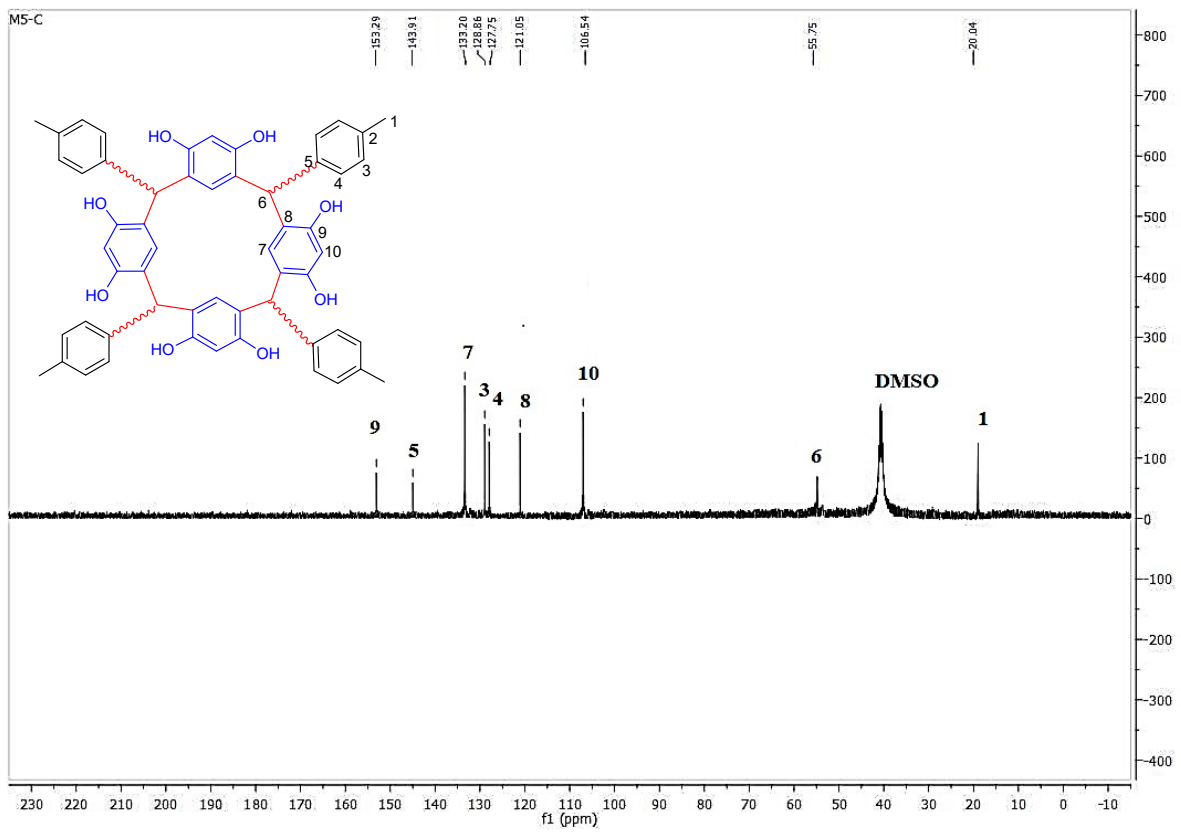
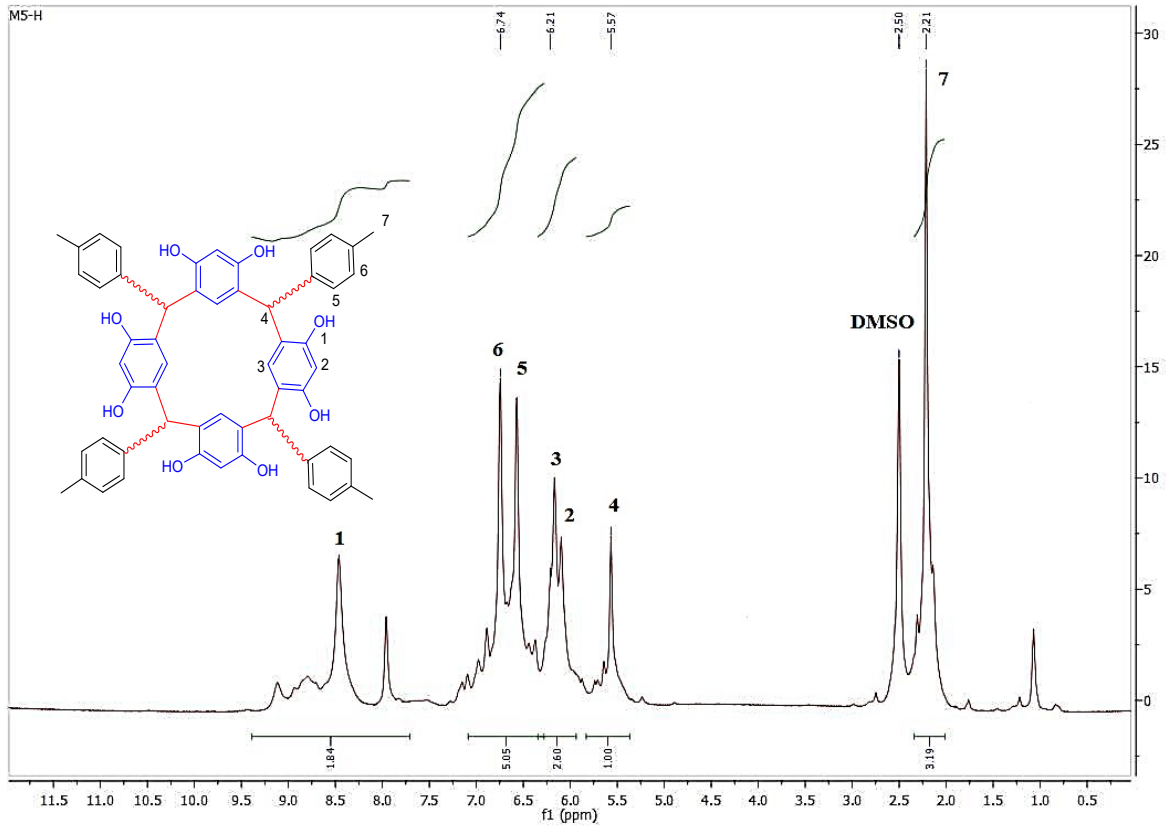


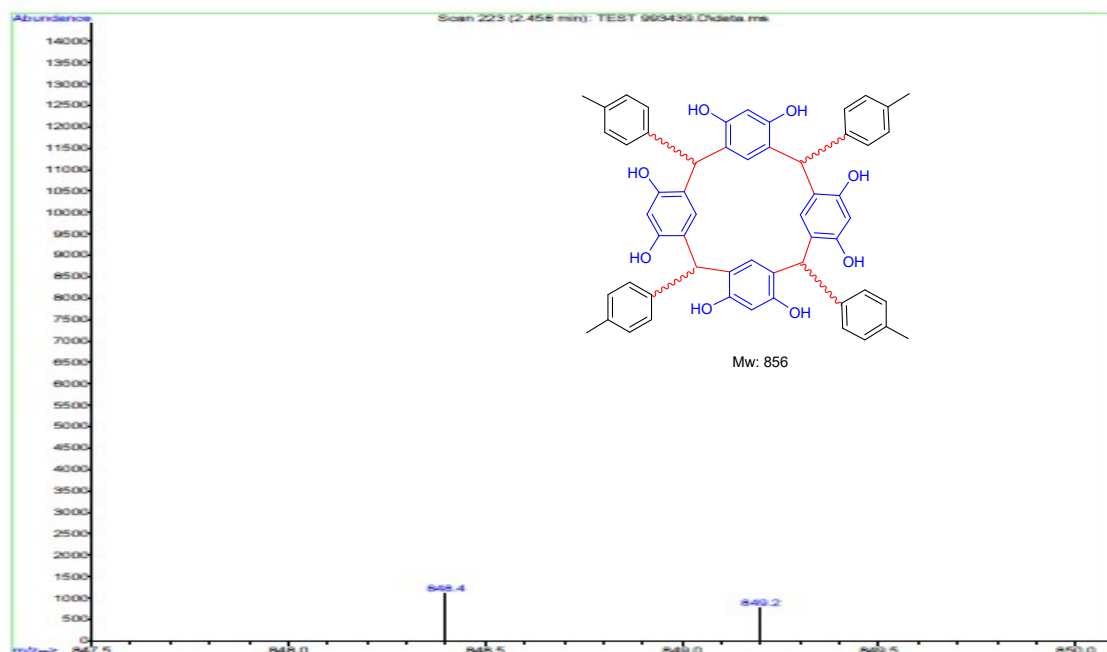
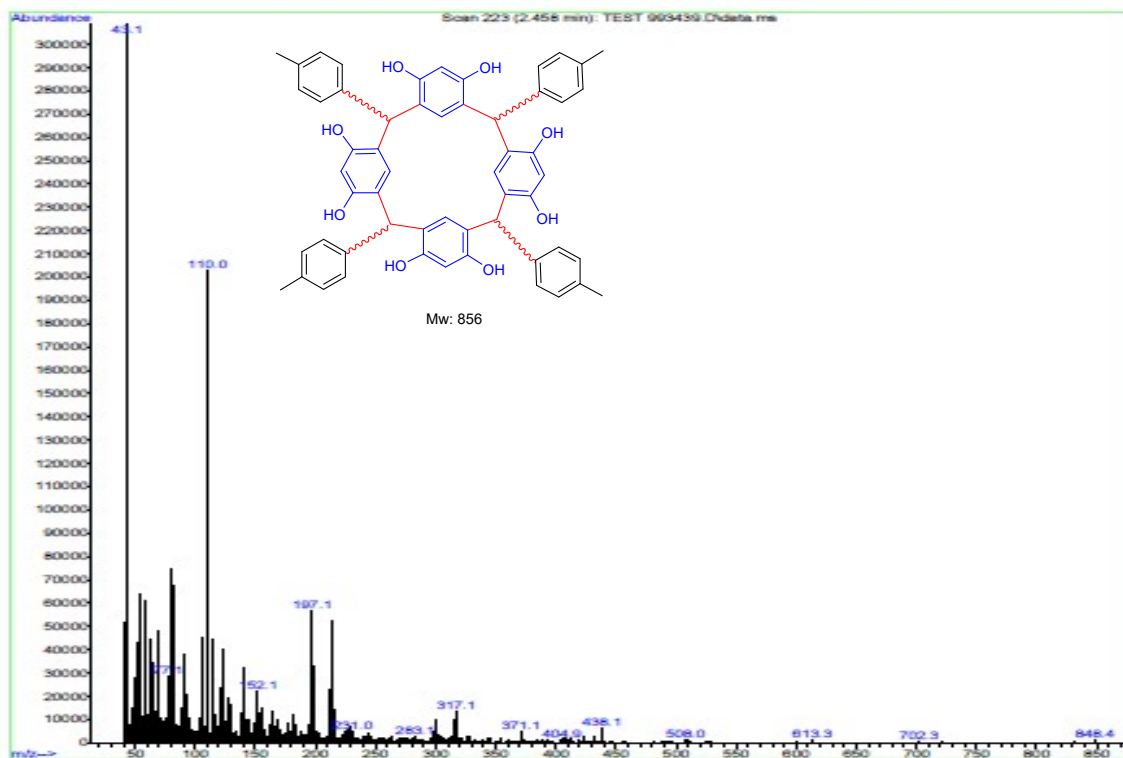


2,8,14,20-Tetra-*p*-tolyl-4,6,10,12,16,18,22,24-octahydroxycalix[4]-resorcarene (3b)

Reddish orange solid, mp > 300 °C (dec); FT-IR (KBr) ν : 3353, 3137, 2919 cm^{-1} ; ^1H NMR (500 MHz, $\text{DMSO-}d_6$) δ ppm: 2.21 (s, 3H, H_7), 5.57 (s, 1H, CH, H_4), 6.09 (s, 1H, Ar-H, H_2), 6.16 (s, 1H, Ar-H, H_3), 6.37-6.97 (m, 4H, Ar-H, H_5 , H_6 , 4H), 7.96-9.12 (OH, broad peak, H_1 , 2H), ppm. ^{13}C NMR (125 MHz, $\text{DMSO-}d_6$) δ ppm: 20.04, 55.75, 106.54, 121.05, 127.75, 128.86, 133.20, 143.91, 153.29. MS: m/z = 856 [M+].



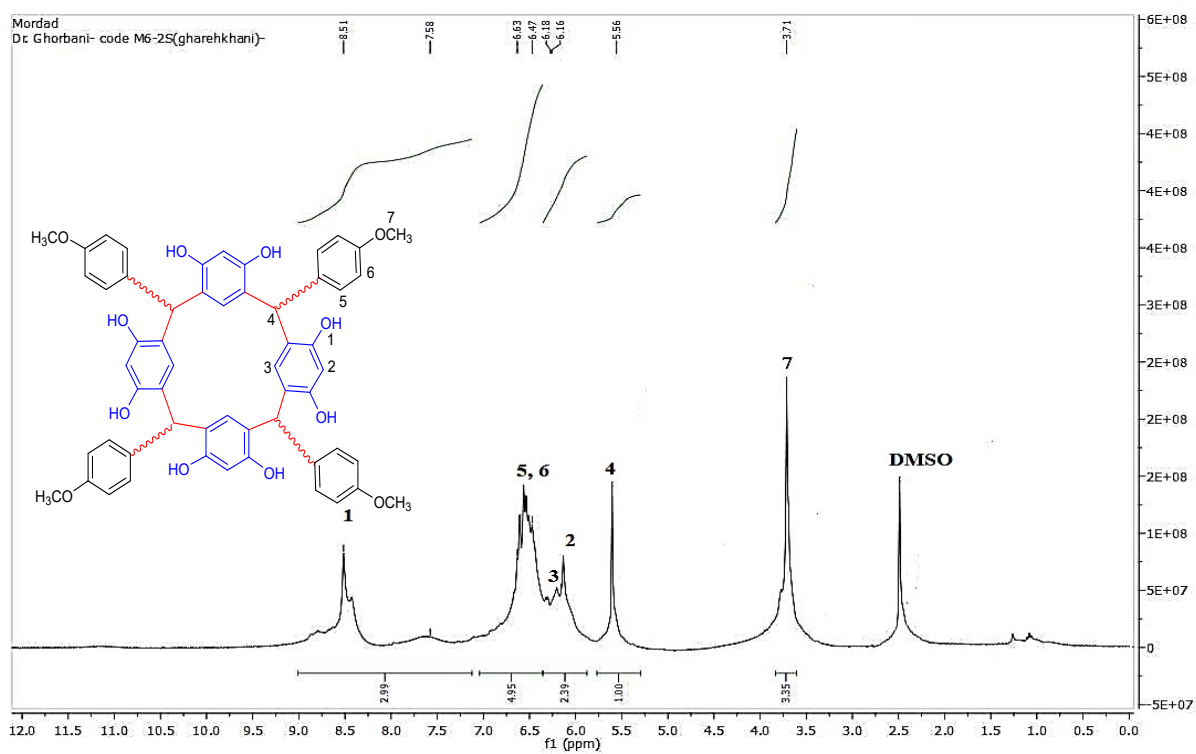
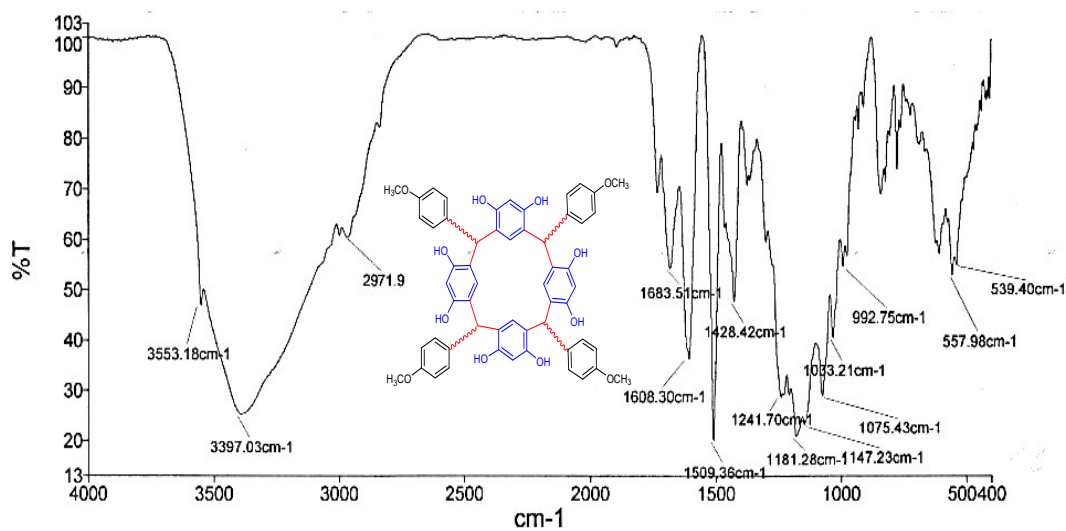


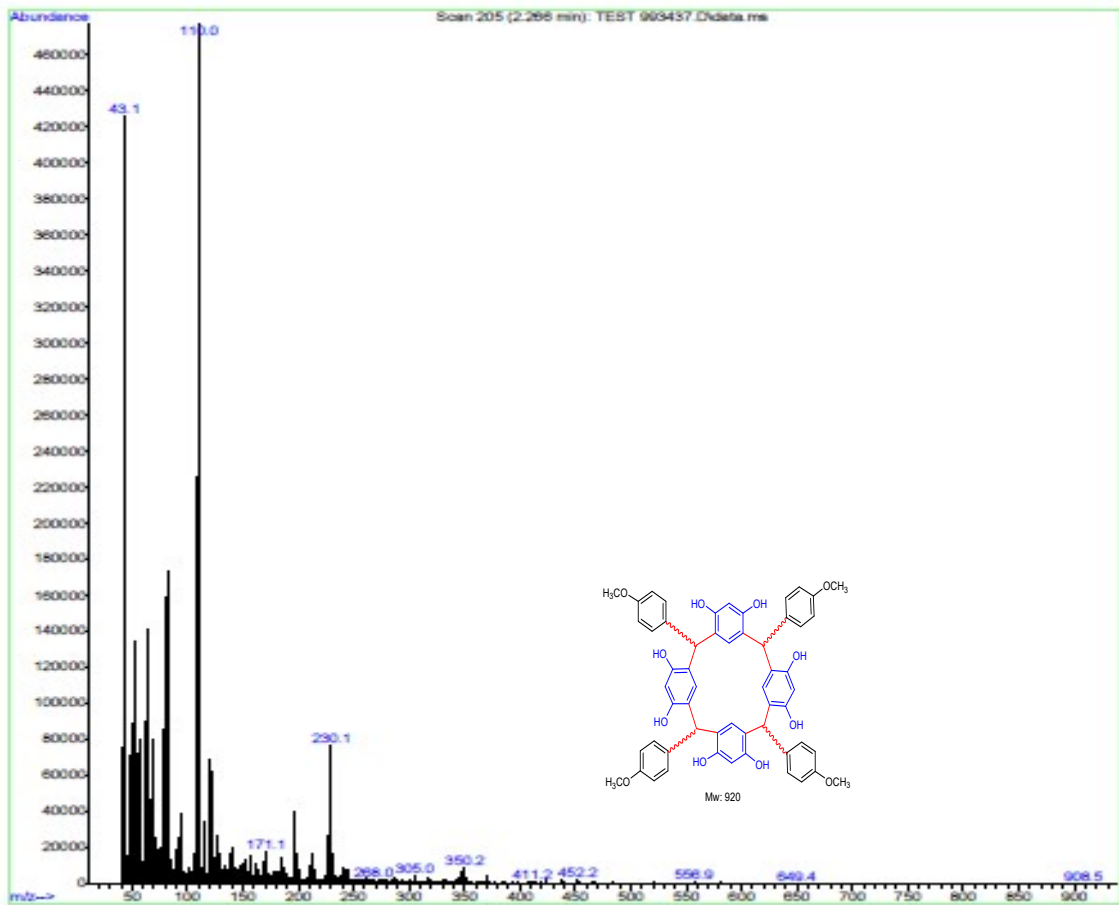
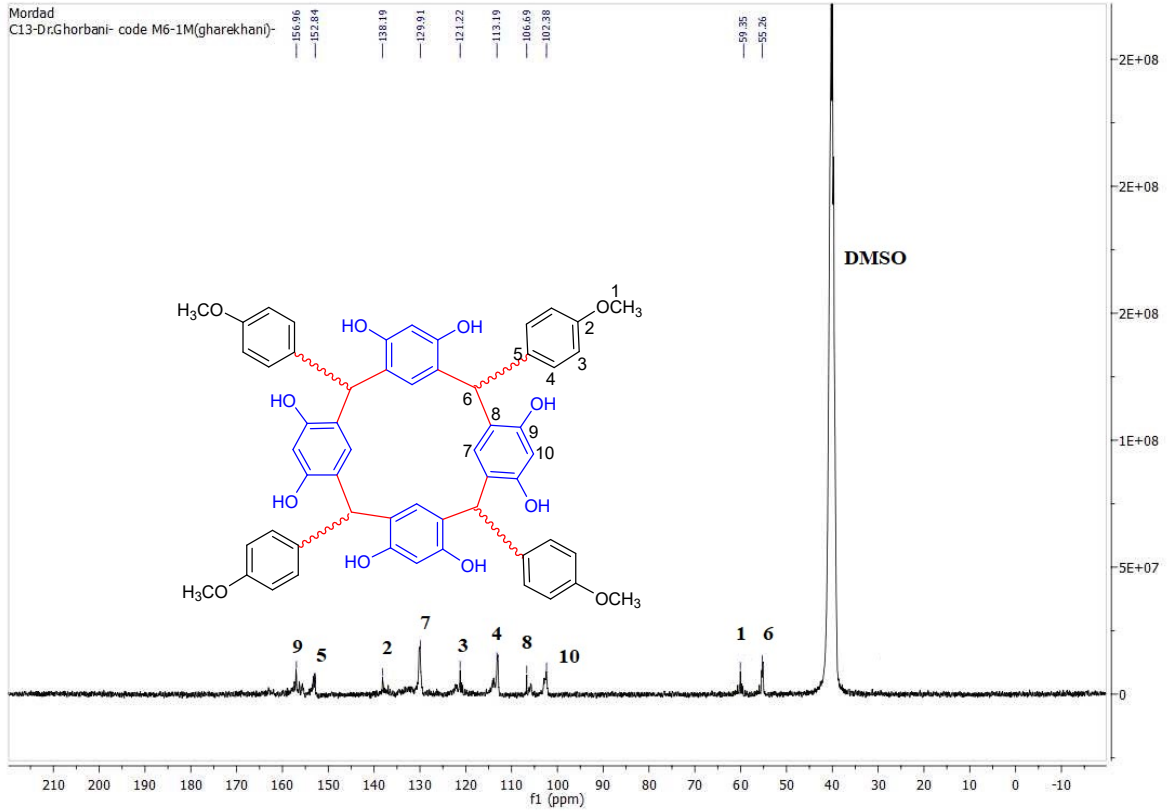


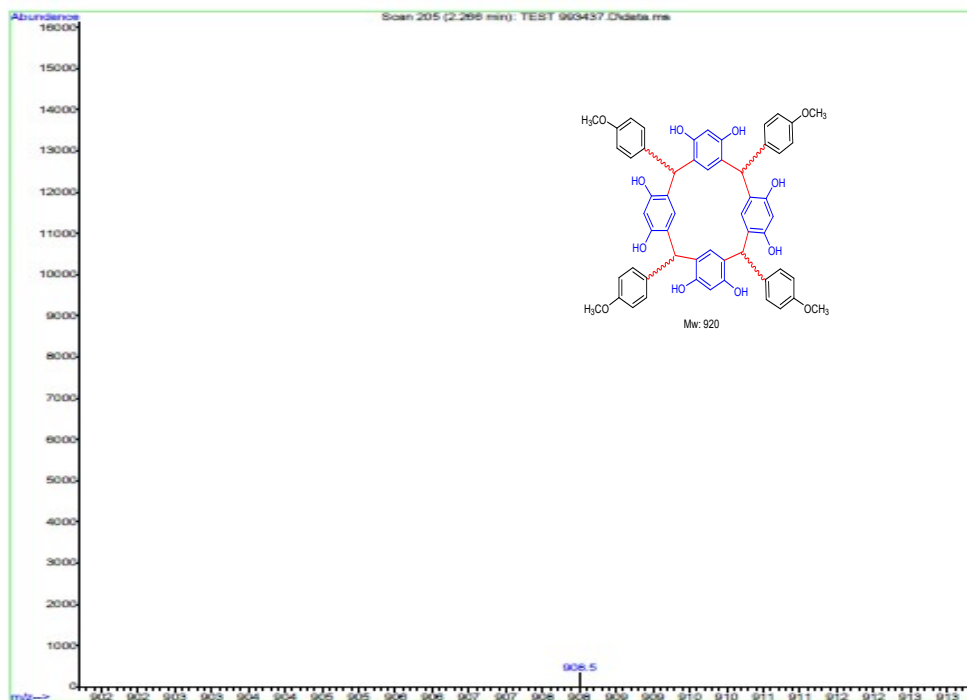
2,8,14,20-Tetra-*p*-methoxyphenyl-4,6,10,12,16,18,22,24-octahydroxycalix[4]-resorcarene (3c)

Reddish orange solid, mp > 300 °C (dec); FT-IR (KBr) ν : 3553, 3397, 2971 cm^{-1} ; ^1H NMR (300 MHz, $\text{DMSO-}d_6$) δ ppm: 3.71 (s, 3H, CH_3 H₇), 5.56 (s, 1H, CH, H₄), 6.16 (s, 1H, Ar-H, H₂), 6.18 (s, 1H, Ar-H, H₃), 6.47-6.63 (m, 4H, Ar-H,

H₅, H₆, 4H), 7.58-8.51 (OH, broad peak, H₁, 3H), ppm. ¹³C NMR (75 MHz, DMSO-d₆) δ ppm: 55.26, 59.35, 102.38, 106.69, 113.19, 121.22, 129.91, 138.19, 152.84, 156.96.

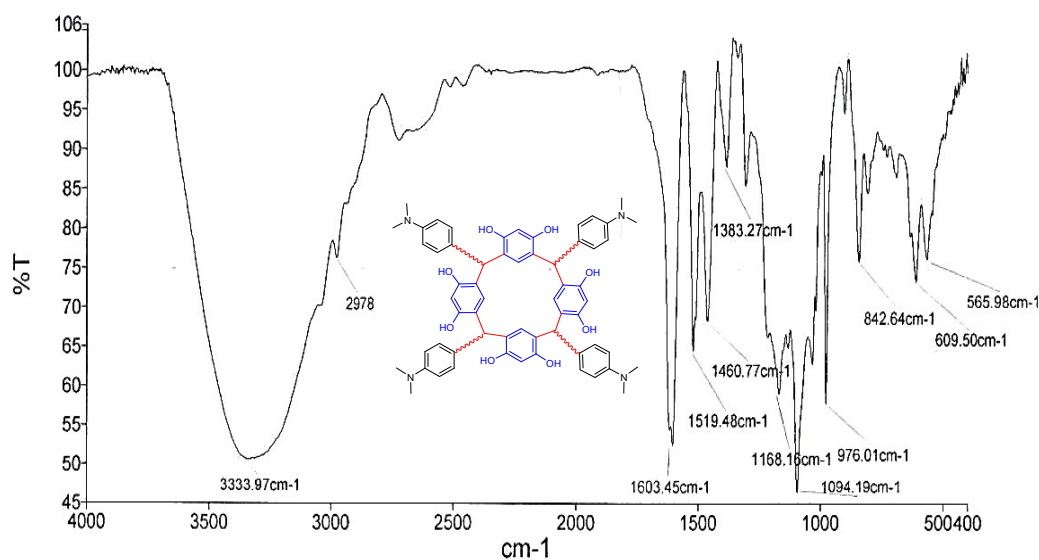


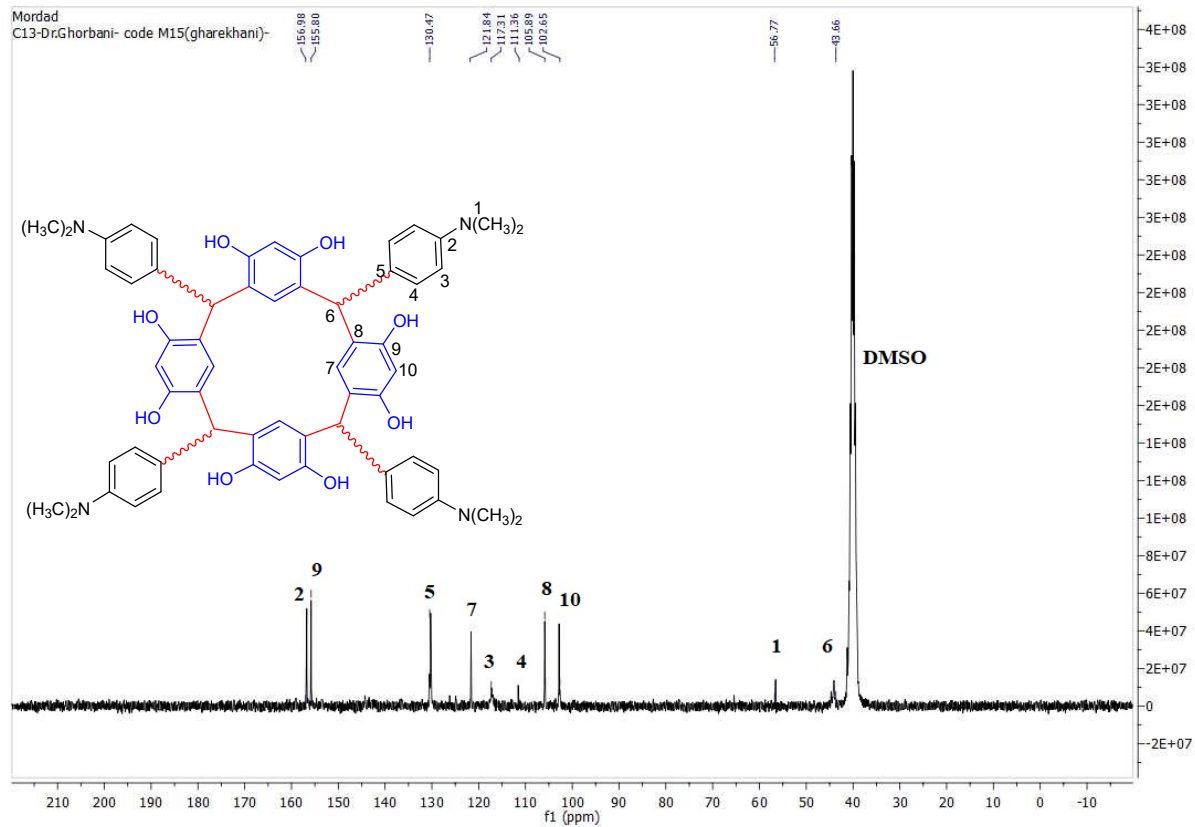
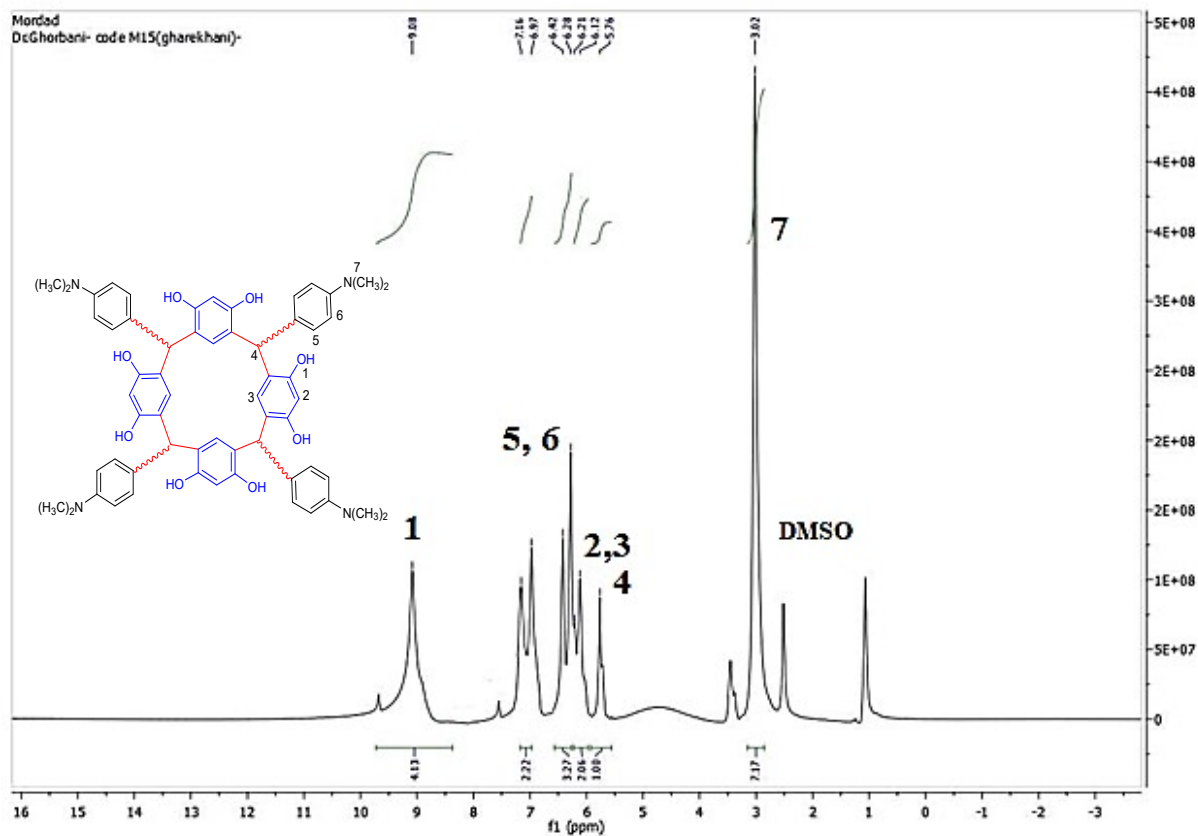




2,8,14,20-Tetra-p-N,N-dimethylphenyl-4,6,10,12,16,18,22,24-octahydroxycalix[4]-resorcarene (3d)

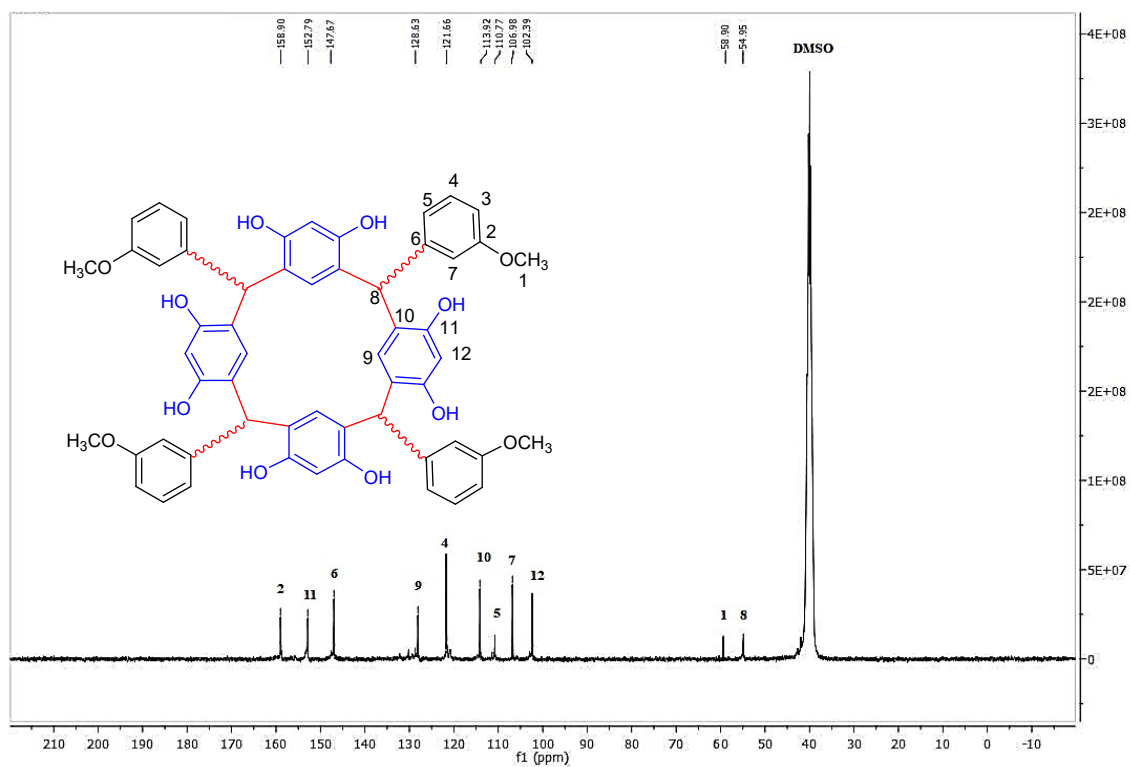
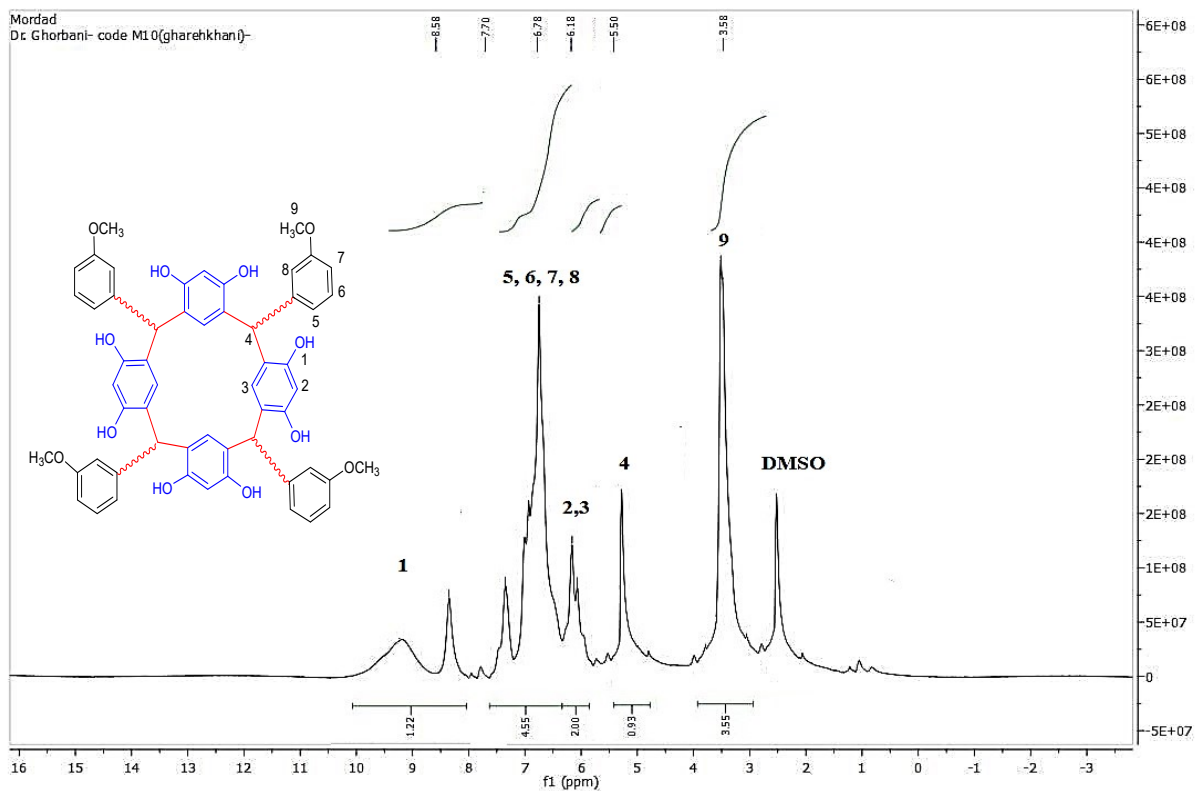
Yellow solid, mp: 228-230 °C; FT-IR (KBr) ν : 3268, 3161, 3004 cm^{-1} ; ^1H NMR (300 MHz, $\text{DMSO-}d_6$) δ ppm: 3.02 (s, 7H, $\text{N}(\text{CH}_3)_2$, H_7), 5.76 (s, 1H, CH, H_4), 6.12 (s, 1H, Ar-H, H_2), 6.21 (s, 1H, Ar-H, H_3), 6.28-7.16 (m, 4H, Ar-H, H_5 , H_6 , 4H), 9.89 (OH, broad peak, H_1 , 4H), ppm. ^{13}C NMR (75 MHz, $\text{DMSO-}d_6$) δ ppm: 43.66, 56.77, 102.65, 105.89, 111.36, 117.31, 121.84, 130.47, 155.80, 156.98.





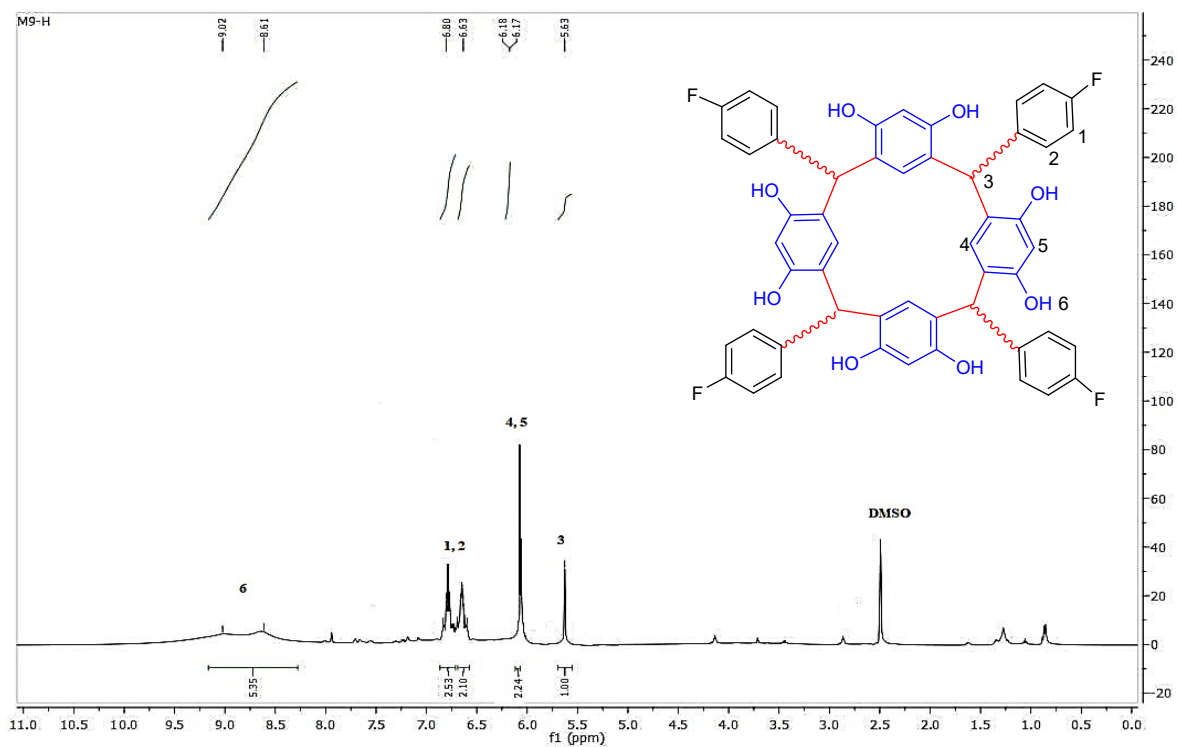
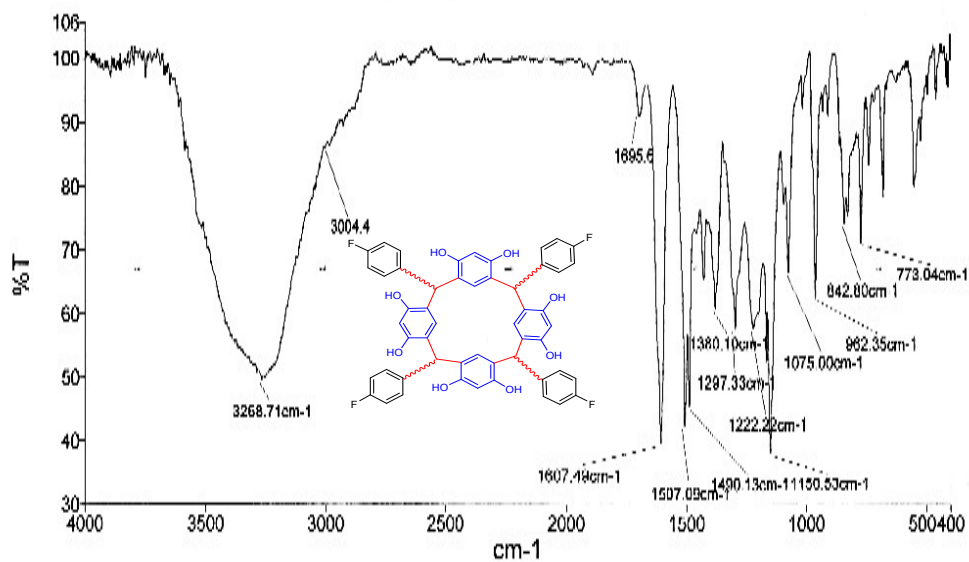
2,8,14,20-Tetra-*m*-methoxyphenyl-4,6,10,12,16,18,22,24-octahydroxycalix[4]-resorcarene (3e)

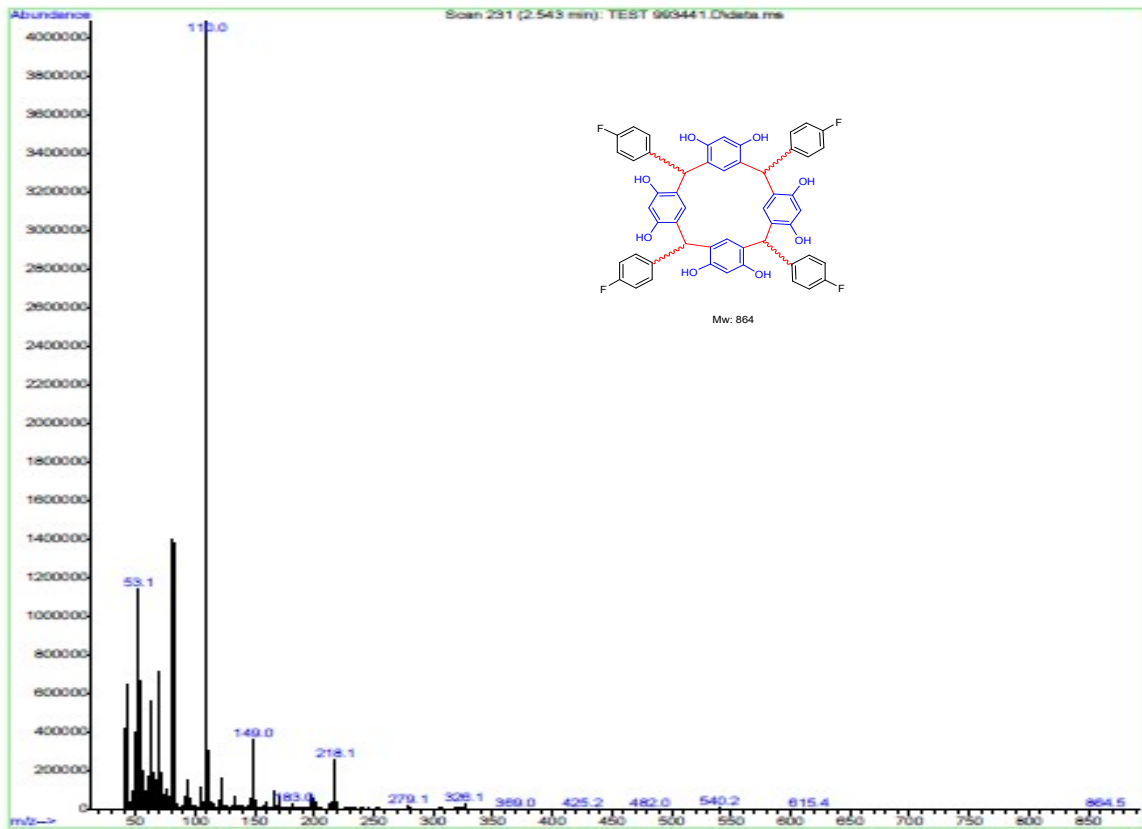
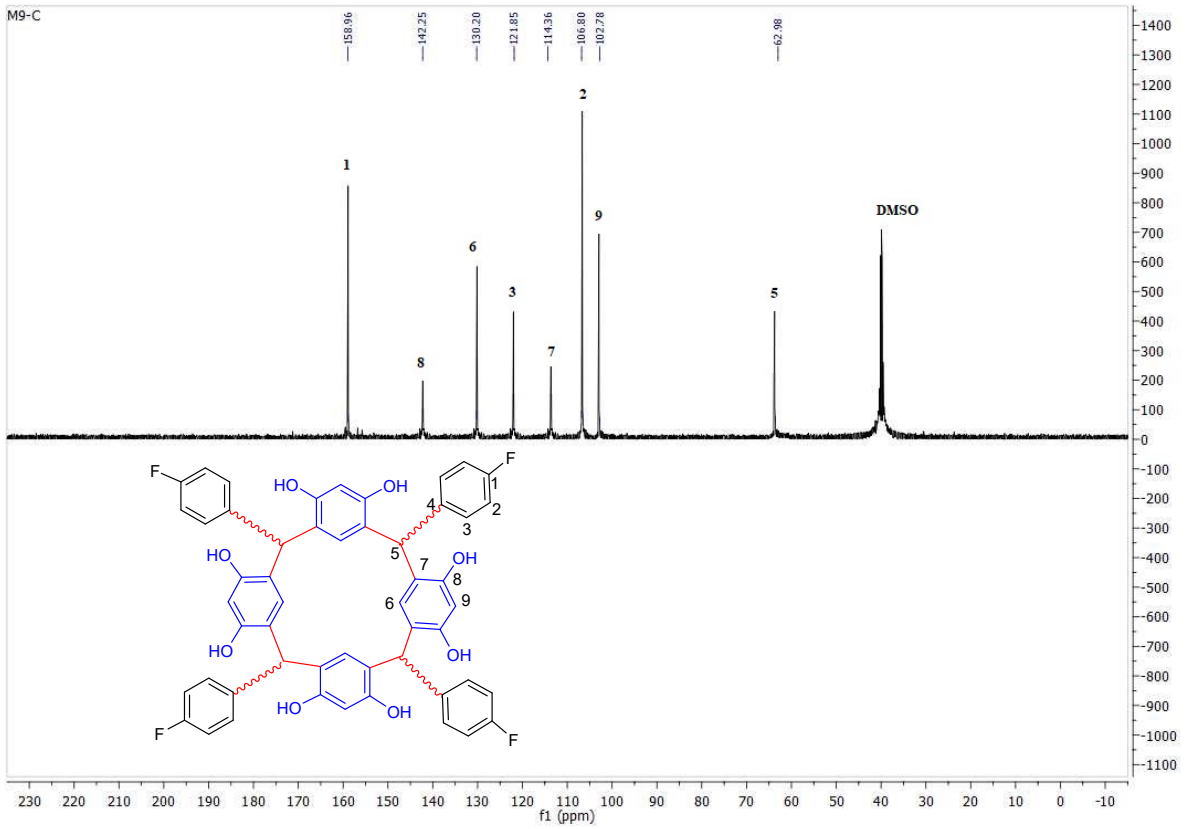
Red solid, mp: 298-300 °C; FT-IR (KBr) ν : 3403, 3059, 2926 cm^{-1} ; $^1\text{H NMR}$ (500 MHz, $\text{DMSO-}d_6$) δ ppm: 3.58 (s, 3H, OCH_3 , H_9), 5.50 (s, 1H, CH, H_4), 6.16 (s, 1H, Ar-H, H_2), 6.18 (s, 1H, Ar-H, H_3), 6.50-7.70 (m, 3H, Ar-H, H_5 , H_6 , H_7 , H_8 , 4H), 8.58-9.5 (OH, broad peak, H_1 , 3H), ppm. $^{13}\text{C NMR}$ (125 MHz, $\text{DMSO-}d_6$) δ ppm: 54.95, 58.90, 102.39, 106.98, 110.77, 113.92, 121.66, 128.63, 147.67, 152.79, 158.90.

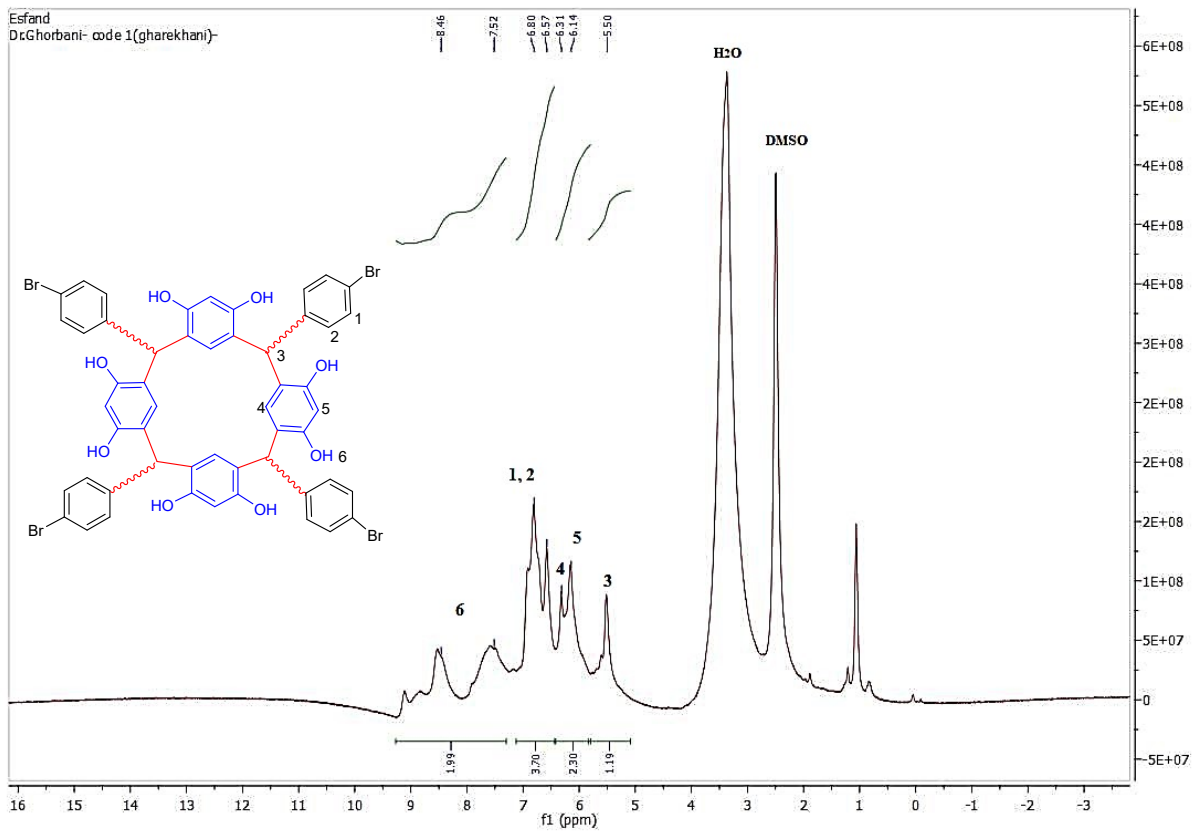
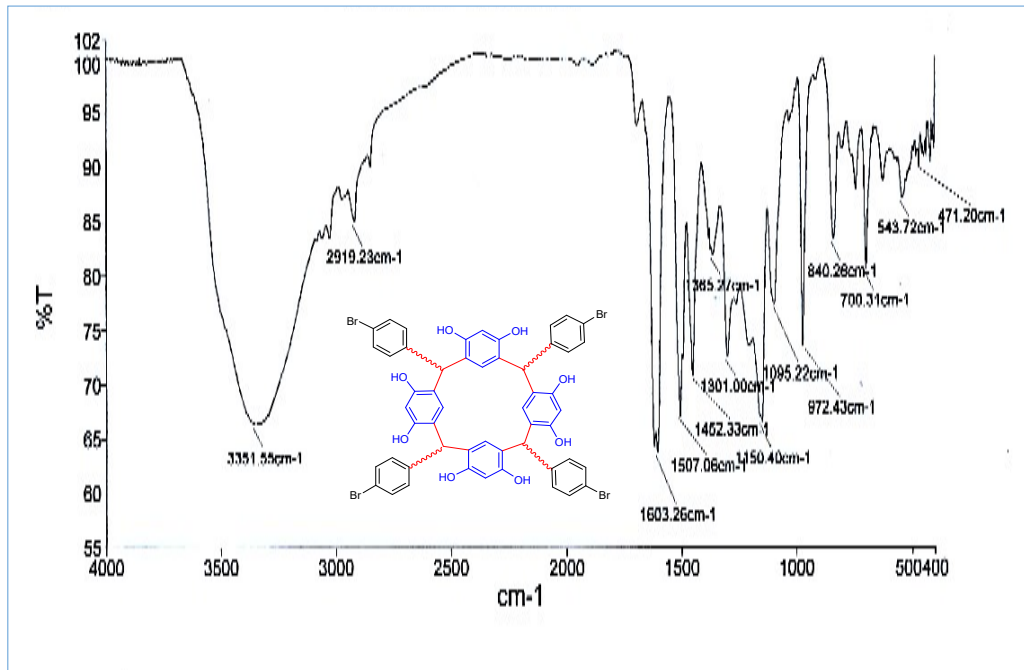


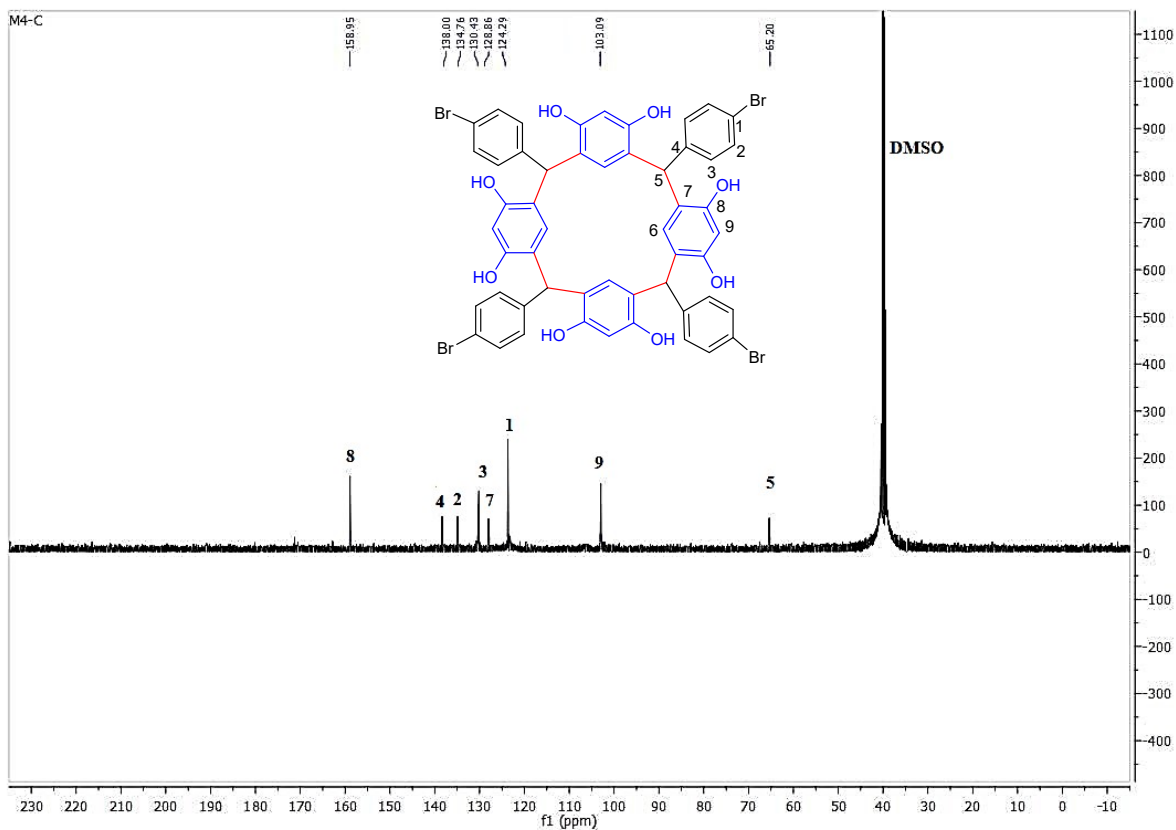
2,8,14,20-Tetra-*p*-fluorophenyl-4,6,10,12,16,18,22,24-octahydroxycalix[4]-resorcarene (3e)

Yellow solid, mp: 223-225 °C; FT-IR (KBr) ν : 3268, 3061, 3004 cm^{-1} ; ^1H NMR (500 MHz, $\text{DMSO-}d_6$) δ ppm: 5.63 (s, 1H, CH, H₃), 6.17 (s, 1H, Ar-H, H₅), 6.18 (s, 1H, Ar-H, H₄), 6.63-6.80 (m, 4H, Ar-H, H₁, H₂, 4H), 8.65-9.05 (OH, broad peak, H₆, 4H), ppm. ^{13}C NMR (125 MHz, $\text{DMSO-}d_6$) δ ppm: 62.98, 102.78, 106.80, 114.36, 121.85, 130.20, 142.25, 158.96. MS: m/z = 864 [M⁺].



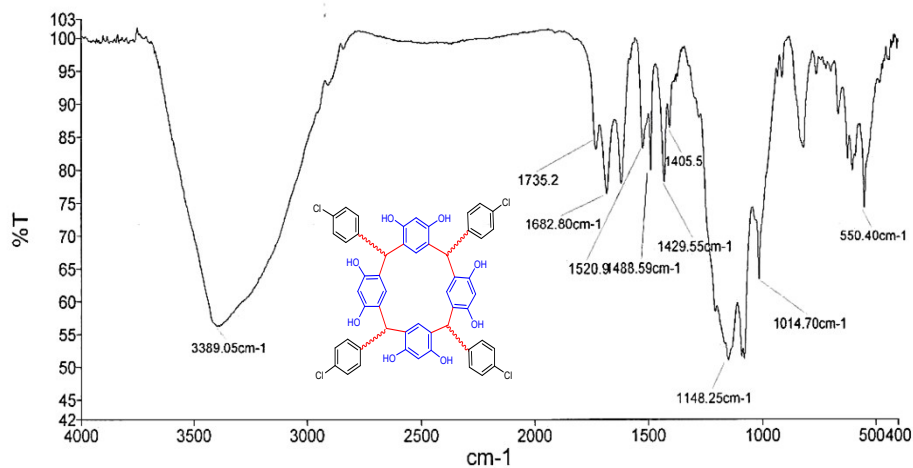


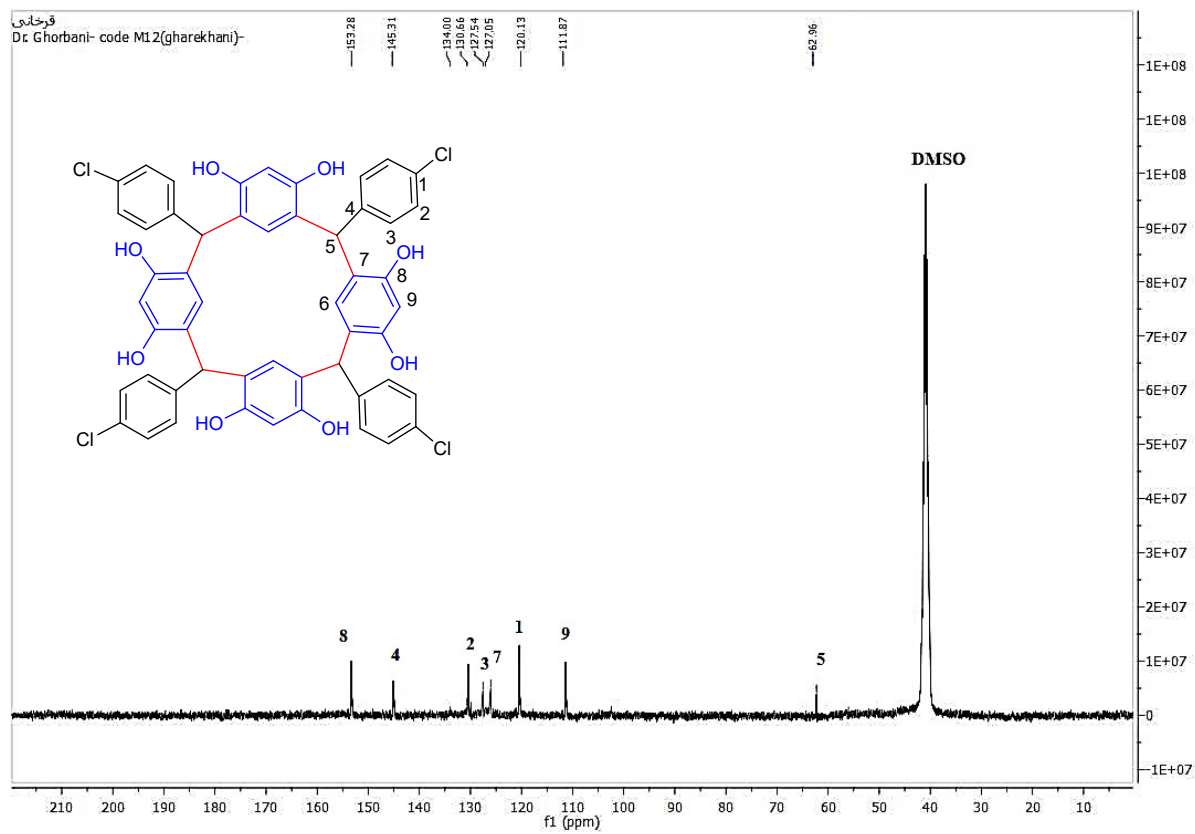
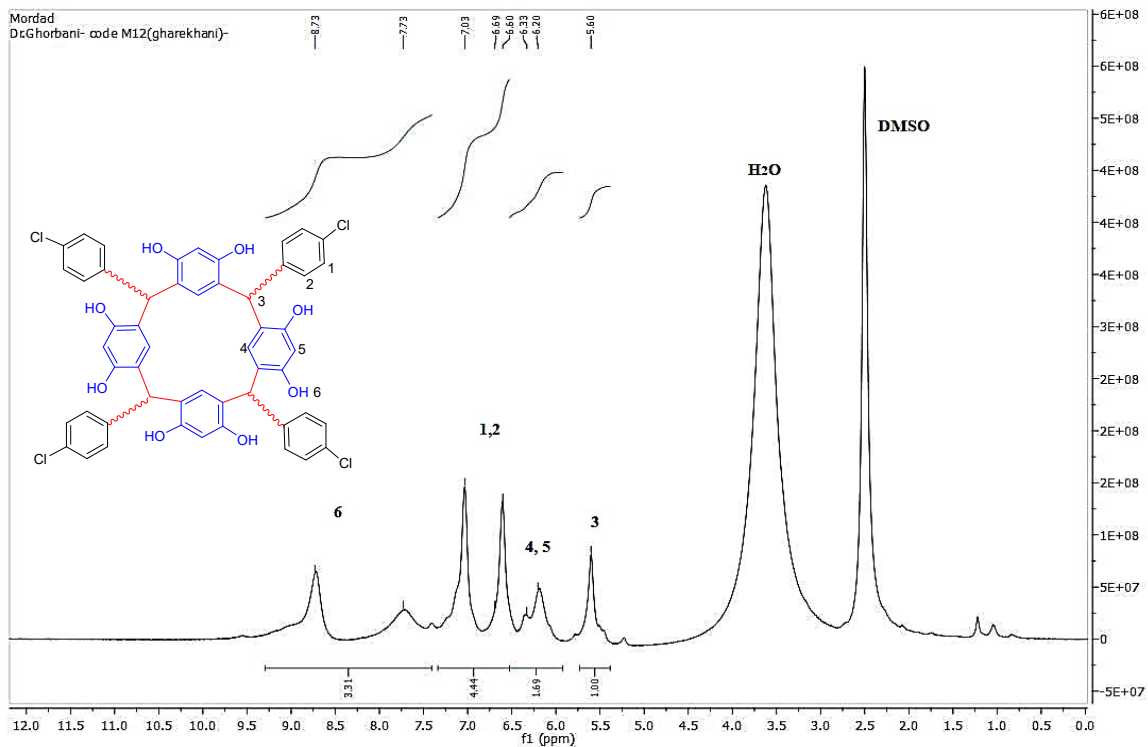




2,8,14,20-Tetra-*p*-chlorophenyl-4,6,10,12,16,18,22,24-octahydroxycalix[4]-resorcarene (3g)

Reddish orange solid, mp: 286-288 °C; FT-IR (KBr) ν : 3389, 1520, 1148 cm^{-1} ; ^1H NMR (300 MHz, DMSO-*d*₆) δ ppm: 5.60 (s, 1H, CH, H₃), 6.20 (s, 1H, Ar-H, H₄), 6.33 (s, 1H, Ar-H, H₅), 6.60 (broad peak, 2H, Ar-H, H₁), 6.69 (broad peak, 2H, Ar-H, H₂), 7.73-8.83 (OH, broad peak, H₆, 3H), ppm. ^{13}C NMR (75 MHz, DMSO-*d*₆) δ ppm: 62.96, 111.87, 120.13, 127.05, 127.54, 130.66, 134.00, 145.31, 153.28.





2,8,14,20-Isobutyl-4,6,10,12,16,18,22,24-octahydroxycalix[4]-resorcarene (3h)

Orange solid, mp: 286-288 °C; FT-IR (KBr) ν : 3258, 2955, 2906 cm^{-1} ; ^1H NMR (500 MHz, DMSO-d_6) δ ppm: 0.83 (s, 3H, CH_3 , H_5), 0.84 (s, 3H, CH_3 , H_8), 1.37 (m, 1H, H_6), 1.67 (m, 2H, H_7), 6.14 (s, 1H, Ar-H, H_4 , 10 Hz), 6.19-6.21 (m, 2H, Ar-H, H_1, H_3), 8.91 (OH, broad peak, $\text{H}_1, 3\text{H}$), ppm. ^{13}C NMR (125 MHz, DMSO-d_6) δ ppm: 15.48, 23.28, 26.19, 32.90, 44.47, 103.09, 122.94, 130.18, 159.19. MS: $m/z = 712$ [M+].

